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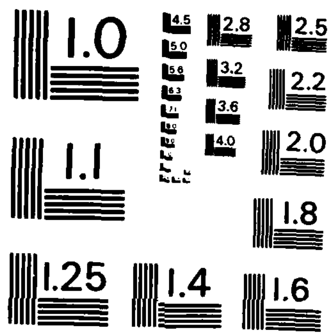

US Army Corps of Engineers

STRUCTURES LABORATORY

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AD-A160 443

THERMAL STABILITY OF CERTAIN HYDRATED PHASES IN SYSTEMS MADE USING PORTLAND CEMENT

by

Alan D. Buck, Jerry P. Burkes, and Toy S. Poole

Structures Laboratory

DEPARTMENT OF THE ARMY
Waterways Experiment Station, Corps of Engineers
PO Box 631, Vicksburg, Mississippi 39180-0631

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As part of the study of hydraulic-cement systems for use in possible underground isolation of nuclear wastes, this study was made to determine the temperature stability of ettringite and chloroaluminate. Either or both of these phases may be expected in a hydraulic cement system depending on the presence of salt (NaCl).

The study of ettringite was made using 15 mixtures that contained portland cement, plaster, 2 levels of water, and in some mixtures, 1 of 6 pozzolans-
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20. ABSTRACT. (Continued)

(3 fly ashes, 1 slag, 1 silica fume, 1 natural pozzolan), plus a 16th mixture with anhydrous sodium sulfate replacing plaster ($CaSO_4 \cdot 1/2H_2O$). Specimens were made and stored at 23°, 50°, and 75° C or 23°, 75°, and 100° C (all four temperatures in one case) for periodic examination by X-ray diffraction for phase composition and ettringite stability, and testing for compressive strength and restrained expansion.

A more limited study of the stability of chloroaluminate was made along the same lines using fewer mixtures, salt instead of plaster, and higher temperatures plus some pressure.

It was found that while some ettringite was decomposed at 75° C, depending on the composition of the mixture, all ettringite was undetectable by X-ray diffraction at 100° C, usually within a few days. The evidence indicates that the ettringite became amorphous and no significant new phases formed in its place. Since there was no corresponding loss in strength or reduction in volume, this loss of ettringite crystallinity was considered to be nondamaging.

Based on much more limited data, chloroaluminate was found to decompose between 130° C at 25 psi and 170° C at 100 psi; no significant phases replaced it.

Preface

This report was prepared for the U. S. Department of Energy (DOE) under modification A008 to contract DE-AI97-81ET 46633. Mixtures were made and testing started later in FY 82 and this testing was continued through late FY 83. An interim report was prepared through 90-day testing in FY 83; that report was expanded to include all of the later testing, revised, and completed in FY 84 as a Milestone under Task 84-5, "Preparation of Topical Reports on Investigations Conducted Prior to FY 83 for Which No Formal Reporting Had Been Done." Mr. Lynn Myers of the Office of Nuclear Waste Isolation (ONWI) was Project Manager when this work started. Mr. Don Moak of ONWI was Project Manager during the bulk of the work. Dr. Roger Wu of DOE-Columbus was Project Manager when the final report was prepared. Mr. Steve Webster of DOE-Columbus was Project Manager when this report was published. The ONWI consultant, Dr. David R. Lankard, provided a technical review of the original draft report that covered the first 90 days of testing.

The report was prepared in the Structures Laboratory (SL) of the U. S. Army Engineer Waterways Experiment Station (WES) under the direction of Mrs. Katharine Mather, former Project Leader, and Mr. Alan D. Buck, present Project Leader. Mr. Bryant Mather was Chief of the SL; Mr. John M. Scanlon, Jr., was Chief of the Concrete Technology Division (CTD). Mr. T. S. Poole of the Cement and Pozzolan Unit calculated the mixture proportions and supervised the making of the mixtures. Mr. R. E. Reinhold (retired) was Chief of the Cement and Pozzolan Unit. Mr. J. P. Burkes and Mrs. J. Ahlvin made and interpreted the X-ray diffraction patterns. This report was prepared by Messrs. Buck, Burkes, and Poole.

COL Tilford C. Creel, CE, and COL Robert C. Lee, CE, were Commanders and Directors of WES during the conduct of this study and the preparation of this report. COL Allen F. Grum, USA, was Director during publication. Mr. Fred R. Brown and Dr. Robert W. Whalin were Technical Directors.

Contents

Conversion Factors, Inch-Pound to Metric (SI) Units of Measurement

Inch-pound units of measurement used in this report can be converted to metric (SI) units as follows:

Multiply	By	To Obtain
Fahrenheit degrees	5/9	Celsius degrees or Kelvins*
inches	25.4	millimetres
pounds (force) per square inch	6.894757	kilopascals
angstroms	0.1	nanometres

* To obtain Celsius (C) temperature readings from Fahrenheit (F) readings, use the following formula: $C = (5/9)(F - 32)$. To obtain Kelvin (K) readings, use: $K = (5/9)(F - 32) + 273.15$.

THERMAL STABILITY OF CERTAIN HYDRATED PHASES IN SYSTEMS MADE USING PORTLAND CEMENT

Introduction

1. Storage of nuclear wastes underground will probably involve the use of at least some systems containing portland cement for filling of shafts, tunnels, and other spaces. Present indications are that these cement-containing systems will need to undergo some degree of expansion after emplacement to produce a tight fit at the contact surface with the host rock. Present belief is that these materials will not be subjected to temperatures much in excess of 100° C at any time. Therefore, there is need for additional information about the thermal stability of compounds such as ettringite and tetracalcium aluminate dichloride-10-hydrate (chloroaluminate) and about what happens to the hydrated cement system if they are altered by heat.

2. While ettringite has been the subject of many investigations and much discussion, these have generally concentrated on different treatments of one phase or cement system (Kalousek and Adams 1951, Jones 1960, Turriziani 1964, Schwiete and Ludwig 1968, Roberts 1968, Mather et al 1978, Taylor and Roy 1980, Ghorab et al 1980, Ogawa and Roy 1981, and Mehta 1972). In general, these indicate that ettringite is stable to about 100° C at atmospheric pressure and that it is usually replaced by tetracalcium aluminate monosulfate-12-hydrate and some form of calcium sulfate when it is destroyed by heat. Higher pressure will raise the decomposition temperature (Ogawa and Roy 1981) and vacuum will lower it (Ghorab et al 1980); Burkes (unpublished) has also found the decomposition temperature is lower in a vacuum. The present work tends to differ from other work in that only a few things were done to a variety of cement systems that might well be used in a nuclear waste isolation sealing project. The determination of compressive strength and restrained expansion along with decomposition temperature is unusual.

3. Considerably less work has been done on the behavior of chloroaluminate in a cement-containing system (Jones 1960, Turriziani 1964, Schwiete et al 1968, Roberts 1968, Lea 1971, and Bensted 1977). The present work with chloroaluminate was abbreviated to meet constraints on available resources and because of repeated equipment malfunctions.

Materials

4. The work was divided into two phases: (a) one dealing with ettringite and (b) one with chloroaluminate.

5. Materials used in the ettringite work were as follows:

   a. Portland cement RC-BCHSR. This was a blend of four different Class H highly sulfate-resistant oil-well cements. Major characteristics are that the caculated C_3A* content is extremely low (3 percent) and the cement is coarse (2100 cm^2/g or 210 m^2/kg).

   b. Plaster AD-661(2 and 3). This was a nonretarded alpha form of $CaSO_4 \cdot 1/2H_2O$. It was used to produce an expansive cementitious system. It was obtained from the Georgia Pacific Co. plant in Blue Rapids, Kansas.

   c. Blending materials. Each of the six materials was used in an amount to replace 30 percent of the cement on a solid volume basis.

      (1) AD-643(2). Granulated blast-furnace slag from the Atlantic Cement Co., Ravena, New York.

      (2) AD-513. Class C fly ash from the Colorado Public Service Co., Pueblo, Colorado (Comanche Plant).

      (3) AD-629. Class C fly ash from Portage, Wisconsin. The supplier was Diversified Concrete of Santa Ana, California.

      (4) AD-628. Class F fly ash from Trona, California. Same supplier as above.

      (5) AD-518. Natural volcanic glass from Hallelujah Junction, California.

      (6) AD-536(3). Silica fume from Reynolds Metal Co., Lister Hill, Alabama.

   d. Each combination of materials was batched and mixed twice. Once with the amount of distilled water to produce normal consistency as determined by ASTM C 187-79. Once with enough water to combine with all of the alumina in the cement and the blending material to form ettringite plus enough additional water to allow for the hydration of the remaining cementitious solids at a water to solids ratio of 0.2.

   e. In addition to the mixture described above, the mixture containing cement, plaster, and fly ash AD-628 with excess water (No. 16) was repeated using reagent grade sodium sulfate (Na_2SO_4) to replace the plaster (No. 18). Compressive strength and length-change tests were made, but X-ray diffraction (XRD) examination was not made.

6. Finally, four mixtures were made using salt (NaCl) to replace the plaster so the expansive product would be chloroaluminate rather than ettringite;

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* C = CaO, A = Al_2O_3.

these were checked by XRD only. These included one with no pozzolan plus ones with ashes AD-513 or AD-628 or slag, all with enough water for normal consistency. The mixture with ash AD-513 (3B(2)) was repeated so strength and expansion tests could be made.

Procedure

Ettringite phase

7. A few preliminary experimental mixtures consisting of portland cement and water; or portland cement, plaster, and water were made and tested for short periods to determine what test temperatures should be used. It was decided that all specimens should be stored for the first 24 hr at 23° C to be followed by continual storage at 23°, at 50°, and at 75° C. During the course of making the mixtures, the 50° C temperature was eliminated and 100° C was added when it was found that 75° C was not always high enough to affect the ettringite. All four temperatures were used for Mixture 17.

8. The combination of cement and plaster with or without one of the six mineral admixtures and two water contents meant that 14 mixtures were required to test all combinations. A total of 15 mixtures were made since the proportions of Mixture (cast) 2 were somewhat in error. No Mixtures 1 or 15 were made because of scheduling problems so the numbering sequence was Mixtures 2 through 14 and 16 and 17. There was also the addition of the mixture (No. 18) substituting sodium sulfate for plaster.

9. Both cubes (ASTM 1983) (2 by 2 by 2 in.) and restrained expansion bars (ASTM 1983) (2 by 2 by 10 in.) were cast from each mixture and Teflon vials were filled (none for Mixture 18) for curing at three temperatures (four for Mixture 17) and testing to 1 year at 12 different ages. The cubes were to be broken for compressive strength, the bars were measured for length change, and the vial samples were examined by X-ray diffraction (XRD). The hardened paste in each vial was removed intact, sawed longitudinally, and cut to a 2-in. length if needed. After minimal grinding with alcohol and abrasive, these sawed surfaces were examined by XRD in an atmosphere of static nitrogen; there was also a small beaker of hot barium hydroxide in the sample chamber to help minimize carbonation during examination. Each sample was examined quickly (2° 2θ/min) from 5 to 60° 2θ on a logarithmic scale; repeated from 5 to about 20° 2θ on a linear 1000 scale to provide quantitative peak intensity data on ettringite,

gypsum, and calcium hydroxide; and examined slowly (0.4^{o} 2θ/min) from 5 to 20^{o} 2θ on a logarithmic scale to provide additional information. The following seven XRD peaks were included within these 15 deg plus new peaks, if present: 9.7-A (ettringite), 7.6-A (gypsum), 7.3-A (unhydrated cement), 5.9-A (unhydrated cement and plaster (?)), 5.6-A (ettringite), 4.9-A (calcium hydroxide), and 4.7-A (ettringite). When it was not possible to make the XRD examination of a vial sample at the scheduled time because of a machine malfunction, the sample was placed in methanol and kept in a freezer until it could be examined. It has been found that this procedure will effectively maintain such a sample for extended periods without significant change. In addition to this routine XRD examination of vial specimens, two special XRD examinations were made. They included:

a. Addition of an internal standard (hornblende) to ground portions of 28-day-old Mixture 14 paste and to 21-day-old Mixture 16 paste. The 9.7-A ettringite peak of these powders was then examined using modified diffractometer settings to provide better determination of this peak position. The experimentally determined ettringite peak position for each paste was then corrected by use of the internal standard hornblende peak at 8.418 A. This was done to determine if there was a detectable XRD difference between ettringite stable at 75^{o} C and ettringite whose crystallinity was destroyed at 75^{o} C.

b. Vial samples of paste Mixtures 12 and 17 were placed in distilled water at about 23^{o} C (73^{o} F) when they were 56 and 42 days old, respectively. Both had been in the 100^{o} C environment and both had had the routine XRD examinations to that time. These two mixtures differ only in water content. These two samples were given limited XRD examination during this new storage condition after 7, 14, 30 days, and about 5-1/2 months. The intent was to see if the ettringite changed at the 100^{o} C storage would reform since recognizable new calcium aluminate sulfate phases had not been formed after initial storage at 100^{o} C.

10. Since the batch size was quite large, there were problems in mixing and overcrowded storage facilities. Therefore, it was necessary to reduce batch size. This was done by reducing the number of cubes broken at each test age from three as specified in ASTM C 109 to two. Such a reduction in cube number results in an increase in the standard deviation of the mean ($s_{\bar{x}}$) of about 20 percent. In some cases, insufficient cubes were available to break two at each age, so only one cube was broken. This reduction represents an increase in the $s_{\bar{x}}$ of about 70 percent over the precision of the strength estimate obtained when three cubes were broken. When such cube shortages occurred,

the single cube breaks were placed at intermediate ages, allowing the strengths at extreme ages to be estimated more precisely.

11. A control mixture was made each time a test mixture was made and specimens from the control batch were tested for strength only. This provided a measure of reproducibility for quality assurance purposes and data were not collected beyond 28 days. Since fewer specimens were needed, this control mixture was made in a smaller mixer. Calculations indicate no significant effect on cube strength due to mixer type. However, the water content of the control mixture was reduced from a water to cementitious materials ratio of 0.27 and 0.28 to accompany Mixtures 2 and 3, respectively, to 0.22 to accompany all other mixtures. Once the 0.22 value was used, this meant the control mixture was then the same as Mixture 12.

12. The cubes and bars were demolded after the first 24 hr; the vials were not demolded until they were used. All of these specimens were kept in plastic bags which were placed in polyethylene containers. The containers which held the bars, cubes, and vials, which were to be stored at elevated temperatures, were immersed in hot water of the desired temperature to within 2 to 3 in. of the top. These containers had plastic lids which tended to become loose fitting because of deformations caused by high temperatures. As a result, there was sometimes standing water in these containers, apparently due to condensation. This water penetrated the plastic bags, consequently, the moisture conditions were not always as intended. This was particularly troublesome with materials stored at 100° C.

13. There was never a significant problem with identification of cubes and bars. However, there was a problem with the vials at ages close to 1 year, especially at the 100° C temperature. Some labels came off at 75° C and additionally some plastic bags were damaged at 100° C so there was a combining of mixtures. This problem was largely solved by study of the contents of the vials in question as immersion mounts with a polarizing microscope plus use of such parameters as color and hardness. Since there was a familiarity with all of the starting materials, this system to check identification worked quite well. There was still some question about Mixture 16 at 75° C which was resolved later by study of the test data.

Chloroaluminate phase

14. As before, a few experimental mixtures were made to select elevated test temperatures in addition to the reference 23° C. Since it was obvious

that the decomposition temperature of the chloroaluminate was above 100° C (Lea 1971), it was necessary to include both temperature and pressure in these considerations. Test temperatures of 130° C and 170° C were selected along with 25 and 100 psi, respectively, for use in autoclaves. Four mixtures were made and vials were filled for periodic XRD examination. No cubes or bars were made from them due to lack of storage space in the two autoclaves. The autoclave that had been set at 170° C failed when the original four mixtures were slightly over 2 weeks old and all of those vials were ruined. In the meantime, one mixture that showed the maximum amount of chloroaluminate was repeated (3B(2)); a few cubes and restrained expansion bars were made in addition to the vials for XRD. In order to have storage space for these specimens, the 130° C vials were placed in moist storage at 23° C and the specimens from the repeat mixture were placed in the remaining autoclave for exposure to 170° C and 100 psi and periodic testing. The temperature rose to about 200° C and the pressure dropped to about 20 psi on the day cubes and bars were to be tested at their 7-day age; complete failure occurred later the same day, so all of these specimens were ruined. The lower temperature vials that had been placed in moist storage were returned to a 130° C and 25-psi autoclave after an interruption of 4 days.

Results

Preliminary experimentation

15. If a vial specimen were quickly placed into an elevated temperature bath after the vial was filled, the vial would distort and an unsatisfactory porous structure would develop in the paste. Since specimens that were given 24 hr curing at 23° C before being subjected to higher temperature did not show these problems, this procedure was used with all of the specimens.

16. XRD examination at early ages of a paste made with portland cement and water showed that ettringite crystallinity was destroyed at a temperature of 75° C. This was the basis for starting the work using 23°, 50°, and 75° C. When other work using portland cement, water, and plaster with or without some of the pozzolans showed that the ettringite was stable at 75° C, this was the basis for changing from 50° to 100° C so the test temperatures were 23°, 75°, and 100° C. However, this change was not made until Mixture 12 was cast.

17. The 16 paste mixtures that were made with plaster and were tested at different ages are shown in Table 1A. Analytical data for the cement, the natural pozzolan, the slag, and the silica fume are shown in Tables 1B, 1C, 1D, and 1E, respectively. Similar data for the plaster (Buck, Burkes, and Reinhold 1981) and the three fly ashes (Buck, Husbands, and Burkes 1983; Buck et al 1983) are given in other WES reports and will not be duplicated here. Quantitative XRD data as peak height intensity of the 9.7-A ettringite peak and the 7.6-A gypsum peak are shown in Tables 2A through 17A with a separate table for each mixture. There were no mixtures numbered 1 and 15 so there are no tables with these numbers. The main item of interest in these tables is to follow the amount of ettringite, as indicated by the 9.7-A peak intensity, as it changes with temperature or age or both for the different combinations of materials. Gypsum ($CaSO_4 \cdot 2\ H_2O$) is the crystalline phase that forms when plaster is combined with water. As long as gypsum is present, more ettringite may form if chemically active alumina (Al_2O_3) and water are still available and the temperature is not too high. Tables 2B through 18B show the average compressive strengths of 1 or 2 or 3 cubes for the same 16 mixtures plus those of the control mixture. These show the effect of temperature and age. As indicated earlier, batch size and thus number of cubes had to be reduced. In addition, there was the usual situation where some specimens are found to be defective on stripping. The combined result of these two situations is that there were not always three cubes to be tested at a stipulated age. Tables 2C through 18C show restrained expansions of individual bars at three different temperatures for the same 16 mixtures. The system of numbering these tables A, B, C, and so on was used so the table number would be the same as the mixture number. The XRD, strength, and expansion data were through 1 year when testing was stopped.

18. Study of the XRD data for the cement and water mixtures and for the other mixtures (Tables 2A through 14A, 16A, 17A) showed:

<u>a</u>. Ettringite was present in all mixtures at all ages when stored at 23° C. For the mixtures without fly ash it increased in amount for periods of 7 to 56 days, depending on the materials, and then remained at a rather constant level thereafter; no effects of water content were found. On the other hand, ettringite continued to increase in amount with age for the three pairs of fly ash bearing mixtures (4, 13; 5, 6; 9, 16) and also with increased water content within each pair of mixtures. Apparently, these fly ashes continue to supply aluminate while the other materials either don't carry it or effectively cease to supply it. Gypsum was also always present and seemed generally steady in amount.

b. Ettringite and gypsum were always present at 50° C.

c. Ettringite crystallinity was destroyed at 75° C when the mixture was just cement and water or also contained plaster and silica fume (Mixtures 10 and 14).

d. Ettringite was not affected at 75° C in any of the other mixtures. This included those with just cement and plaster and those with one of the three fly ashes or the natural pozzolan or the slag. Gypsum was almost always present in the mixtures at 75° C.

e. Ettringite crystallinity was destroyed at 100° C; however, this sometimes required up to 7 or 14 days to be accomplished. Gypsum was also destroyed at 100° C and seemed to reappear in its anhydrous form (anhydrite). No significant new phases were found that could be ascribed to change in the ettringite. Since ettringite detectable by XRD disappeared and no direct replacement alteration compound such as tetracalcium aluminate monosulfate-12-hydrate (monosulfoaluminate) replaced the ettringite, its crystallinity was destroyed by temperature and it is no longer ettringite in the sense of being a crystalline compound identifiable by XRD. As indicated later, it did not reform when given a more favorable environment.

f. There was a dramatic increase in amount of ettringite with temperature (<100° C) when fly ash was present. This was taken to show that the ash was continuing to provide alumina for the formation of more ettringite and that this was faster at higher temperature as might be expected.

g. When silica fume was present as in Mixtures 10 and 14, the calcium hydroxide (CH) was usually gone or much reduced at both higher temperatures by the 48-hr age; it was then completely gone at all temperatures after a few more days. Apart from this striking behavior with the fume mixtures, there was also some reduction in amount of CH with increasing temperature, especially in the mixtures containing the natural pozzolan and the fly ashes. These data are not in the tables.

h. There were seven pairs of mixtures that differed only in water content. These were 12 and 17, 3 and 7, 4 and 13, 6 and 5, 11 and 8, 9 and 16, and 14 and 10. Each pair is listed by increasing water content. Comparison of the ettringite content in the four pairs of mixtures treated at comparable temperatures (12 and 17, 3 and 7, 11 and 8, and 6 and 5) indicated that there were no consistent or significant differences for the pairs without fly ash (12 and 17, 3 and 7, 11 and 8). However, Mixtures 6 and 5 with ash AD-513 showed the same sort of trend in ettringite at 50 and 75° C as mentioned earlier for 23° C (i.e., increase with age and water content).

19. The XRD examination of the 9.7-A peak of ettringite crystallinity lost at 75° C (Mixture 14) and ettringite stable at 75° C (Mixture 16) for more accurate measurement by use of an internal standard gave the following results:

	Ettringite Peak Corrected With Internal Standard
Mixture 14 (Unstable)	9.704 A
Mixture 16 (Stable)	9.710 A

This difference of 0.006 A may be real but this low value from a single determination is not adequate to prove that the difference in temperature stability is directly attributable to a crystal lattice modification.

20. The periodic XRD examination of XRD samples (Mixtures 12 and 17) whose ettringite crystallinity had been destroyed by 100° C temperature showed the following after these samples had then been kept in distilled water at room temperature for 7, 14, 30 days, and 5-1/2 months:

a. A small amount of ettringite had reformed after 7 days, but it did not increase significantly with additional storage time.

b. Gypsum crystals up to 1 cm in length precipitated on the surface of the samples. They presumably came from the anhydrite that always formed in samples heated enough to destroy gypsum usually present at 75° C (Tables 2A through 11A).

c. Hydrogarnet was already present when these samples were placed in water and persisted over the time of storage. The presence of this aluminum-bearing compound may have precluded formation of new ettringite because no aluminum was available for it.

21. In general, the XRD results beyond 90-days age were consistent with those to the 90-days age. Temperatures to 50° C had no significant effect. The 75° C temperature was selective in that it destroyed the crystallinity of some ettringite, and 100° C destroyed the crystallinity of all of the ettringite even though it sometimes took a few days for this to happen. There was also a tendency for the formation of some hydrogarnet or sometimes tetracalcium aluminate hemicarbonate-12-hydrate (hemicarboaluminate) or both plus a tendency for the calcium silicate hydrate (CSH) to improve in crystallinity. A peak at 12+ to 13+ Angstroms tended to show at 100° C; this suggested an increase in crystallinity of the CSH. However, none of these can be construed as a direct replacement of the ettringite that lost its crystallinity at this temperature.

22. Tables 2B through 14B along with Tables 16B, 17B, and 18B show compressive strength data. Generally, these data are as expected with strength increasing with age and with temperature. In addition, there are a few cases where it seems obvious there was an error of some sort. For example, in Table 13B in the 100° C row, a strength of 8070 psi at 28 days in between

strengths of 2890 and 2810 psi at 21 and 56 days, respectively, is indicated. Additionally, the reduction in batch size plus elimination of obviously defective cubes which meant that three cubes could not always be tested plus the scattered moisture variations mentioned earlier will explain some of the data.

23. There is no indication that temperature adversely affects compressive strength for temperatures as high as 75^{o} C. However, three of the five mixtures cured at 100^{o} C substantially failed to gain as much strength as the same materials cured at 75^{o} C (Mixtures 13, 16, and 17). The failure of these mixtures to gain as much strength as when cured at 100^{o} as when cured at 23^{o} and 75^{o} does not appear to be related to the loss of crystallinity of the ettringite. The loss of crystallinity of the ettringite occurred at very early ages in all five mixtures, but low strength gain became apparent at later ages in the three mixtures in question and not at all in the other two mixtures cured at 100^{o}. Thus, although data are incomplete because of the changes in the experimental design, it appears as though low strengths for materials cured at 100^{o} were due to factors other than loss of crystallinity of the ettringite. It is possible that low strength development in these mixtures is a result of rapid formation of ettringite at elevated temperatures which produced cracks due to expansion. This could plausibly tie in with the fact that Mixtures 13, 16, and 17 would be lower early strength because of the presence of fly ash (13, 16) or higher water content (17). The general phenomenon of reduced strength development at elevated temperatures is well documented in the literature, along with probable factors underlying it (Lea 1971, pp 397-398; Smith 1978, Mindess and Young 1981, p 530; Carette et al 1982), consequently it will not be dealt with further here as it is beyond the scope of this report.

24. Restrained expansion data are shown in Tables 2C through 14C and Tables 16C, 17C, and 18C. The 1-day values are for three bars cured at 23^{o} C. The values thereafter are for single bars stored at different temperatures. Replication at each temperature was not feasible because of inadequate storage space. Thus, the only opportunity to evaluate the size of the experimental error in these data is through analysis of the 1-day data. The standard deviation of the percent expansion at 1 day is 0.018 percent. This estimate of experimental error is probably a minimum figure because of unintended moisture variations and probable changes in the tensile properties of the restraining rods with age. This level of error is large relative to the levels of expansion observed in the various mixtures, consequently, in the absence of

replication, detailed interpretations of the expansion data are not warranted. A thorough quantitative description of the expansive behavior of these mixtures would require a more elaborate experimental treatment, which was beyond the scope of this program. Nonetheless, comparison of XRD data for ettringite with expansion data for matching mixtures at 75° C and 100° C (12, 13, 14, 16, 17) seem to show that the absence of crystalline ettringite had no detrimental effect on expansion; this is considered particularly significant in view of the anticipated usage of grouts or concretes somewhat simulated by these mixtures. Shrinkage or inhibited expansion of mixtures containing as much replacement of silica fume for cement (30 percent by solid volume) as Mixtures 10 and 14 is typical and should be noted in case this material is seriously considered for use. On the other hand, the use of 5 or 10 percent silica fume replacement for cement, while still expansion inhibiting, may be useful. This can be seen for Mixtures M-9-D, E, F in Table 5 of (Buck et al 1983). Finally, it should be noted that increased temperature per se did not mean an automatic decrease in expansion under moist storage conditions (Tables 2C through 14C, 16C, 17C, 18C).

Chloroaluminate

25. The work to determine the stability of chloroaluminate was done in a similar manner to that done to determine ettringite stability. The differences were:

a. There were 4 different mixtures instead of 16. One mixture was made twice to include strength and restrained expansion data.

b. Salt was used in place of plaster.

c. Elevated temperatures were intended to be 130 and 170° C. Since these are above the boiling point of water, it was necessary to include pressure as a factor. The pressures with these salt mixtures were 25 and 100 psi instead of 1 atmosphere.

26. Information about the mixtures is given in Table 1A(2).

27. XRD data for ettringite and chloroaluminate peak intensities (9.7-A, 7.8-A) are shown in Tables 19 through 23. Preliminary work with several experimental mixtures had indicated that chloroaluminate was destroyed at 170° C and 100 psi but not at 130° C and 25 psi; this is generally verified by these tables. Apparently, there was not enough salt in Mixture 1B (Table 19) to form chloroaluminate. Mixture 3B (Table 21) formed the most chloroaluminate; this is the reason this mixture was made again to include compressive strength data (Table 24) and restrained expansion data (Table 25). Due to the failure of the autoclaves with subsequent overheating and some drying of the original 170° C

samples and the 170° C mixture 3B(2) samples, only the XRD data are of much value. Autoclaving tended to increase the crystallinity of the calcium silicate hydrate as would be expected. As with the plaster mixtures, there was less CH with increasing temperature.

Discussion

28. Since the mixtures or pairs of mixtures each constitute a different system and there are different kinds of data along with variables of temperature, age, and water content or pressure in some cases, detailed study is required in each case to obtain maximum results.

Ettringite

29. An interesting result is that there is not a constant temperature for the alteration of ettringite in these different pastes. When the paste is only portland cement and water or portland cement, water, plaster, and silica fume (Tables 10A, 14A), ettringite crystallinity is usually wholly or largely destroyed* at 75° C. With all of the other mixtures of cement, plaster, and water or cement, plaster, water, and one of the five other mineral admixtures (three ashes, one slag, one natural pozzolan), ettringite was still present after exposure to 75° C through 1 year.

30. It is known from some of the present references and from other literature (Diamond and Lochowski 1983) that ettringite need not be pure; iron or silica can substitute for aluminum and perhaps hydroxyl or carbonate ions can substitute for sulfate ions. Often compositional change in a crystalline phase is accompanied by a systematic shift in XRD spacings. Unfortunately, such changes in ettringite do not appear to produce significant changes as discussed earlier. This effort indicated the position of the XRD peak for the unstable ettringite in Mixture 14 was 9.704 A while it was 9.710 A for stable ettringite in Mixture 16. While this difference of 0.006 A could be significant, it was not considered conclusive in this case. Nonetheless, the most likely answer to the difference in stability of ettringite with temperature still appears to be slight differences in composition of the ettringite.

31. As mentioned earlier, there was some concern about identification of a mixture No. 16 XRD vial at 75° C. Examination of the XRD data in Table 16A

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* No lorger detectable by XRD.

does not show anything that is considered a significant change so it is believed the questionable sample was correctly identified.

Conclusions

32. The literature mentioned earlier generally indicates that ettringite is destroyed at or about 100° C. This work confirmed that value. In addition, the most striking finding was that some ettringite became undetectable by XRD at 75° C while other ettringite was similarly affected at 100° C. It is believed that the difference is due to its purity with impure ettringite being stable to the higher temperature.

33. The most significant finding, established by study of ettringite levels by XRD and compressive strength or expansion levels to 1 year, was that this loss of detectable ettringite was not reflected in detrimental effects to the strength or volume of the paste mixtures that were used. This is significant for repository sealing considerations because it largely removes the concern about detrimental effects of temperatures to 100° C on candidate mixtures based on portland cement systems.

34. Subsidiary findings about ettringite included:

a. If a long-term source of aluminum is available, as for example from certain fly ashes and there is an ample supply of calcium, sulfate, and water then ettringite will continue to form at least to 1 year. In these cases more ettringite formed at higher water contents. Apart from these special cases additional water did not usually result in more ettringite.

b. The use of portland cement and plaster with a variety of individual mineral admixtures (three ashes, one slag, one natural pozzolan, one fume) or without these admixtures demonstrates the range of materials combinations that appears to be viable candidates for repository sealing usage.

c. Once the ettringite was converted to a noncrystalline form by heat, there was little tendency to reform under moist storage conditions at room temperature.

d. While monosulfoaluminate was expected to replace ettringite, this did not happen. The new phase that did tend to form after heating to 100° C was hydrogarnet; it was not considered a usual conversion product of ettringite.

35. Although the study of the stability of chloroaluminate was beset by numerous difficulties, it was found that this compound decomposed between temperatures and pressures of 130° C and 25 psi to 170° C and 100 psi. The strength

and length-change data were not considered useful due to autoclave failures. As in the case of ettringite, no significant new phases were formed. Since there was no recognizable deleterious effect on paste strength or volume due to removal of crystalline ettringite, it seems likely the same should also be true for removal of crystalline chloroaluminate by temperature.

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Table 1A

Composition and Casting Dates of Phase A Pastes*

			Materials			
			Admixtures		Water	
Mixture (Cast) No.**	Casting Date	Cement (RC-BCHSR) %	Plaster (AD-661(2,3)), %	Other, %	Amount	Water to Cementitious Solids Ratio
2	3 June 82	81.3	18.7	--	Excess	0.32
3	7 June 82	59.2	16.8	24.0 slag (AD-643(2))	Normal consistency (n.c.)	0.28
4	9 June 82	57.6	20.8	21.6 ash (AD-629)	n.c.	0.28
5	11 June 82	53.4	27.4	19.2 ash (AD-513)	Excess	0.48
6	15 June 82	53.4	27.4	19.2 ash (AD-513)	n.c.	0.35
7	17 June 82	59.2	16.8	24.0 slag (AD-643(2))	Excess	0.36
8	21 June 82	59.0	21.6	19.4 nat. pozz. (AD-518)	Excess	0.43
9	23 June 82	56.7	25.7	17.6 ash (AD-628)	n.c.	0.34
10	25 June 82	69.5	9.3	21.2 silica fume (AD-536(4))	Excess	0.32
11	29 June 82	59.0	21.6	19.4 nat. pozz. (AD-518)	n.c.	0.36
12	1 July 82	90.8	9.2	--	n.c.	0.22
13	5 July 82	57.6	20.8	21.6 ash (AD-629)	Excess	0.43
14	8 July 82	69.5	9.3	21.2 silica fume (AD-536(4))	n.c.	0.30
16	13 July 82	56.7	25.7	17.6 ash (AD-628)	Excess	0.46
17	15 July 82	90.8	9.2	--	Excess	0.34
18†	10 August 82					

* Made with cement and plaster or cement, plaster and 30 percent by solid volume of one of six mineral admixtures. Made once with enough water for normal consistency and again with more water to make all possible ettringite from all alumina and still have enough water for 0.2 ratio of water to cementitious solids.

** No mixtures 1 or 15 were made.

† Same as Mixture 16 with sodium sulfate in place of plaster.

Table 1A(2)

Composition and Casting Dates of Phase B Pastes

Cast No.*	Casting Date	Materials				Water to Cementitious Solids Ratio
		Cement (RC-BCHSR), %	Admixtures		Water Amount	
			Salt (AD-640), %	Other, %		
1-B	31 Aug 82	95.9	4.1	--	Excess	0.24
2-B	31 Aug 82	69.4	9.1	21.5 ash (AD-628)	Excess	0.29
3-B	1 Sep 82	66.4	9.8	23.8 ash (AD-513)	Excess	0.30
3-B(2)	16 Sep 82	66.4	9.8	23.8 ash (AD-513)	Excess	0.30
4-B	1 Sep 82	67.1	5.8	27.1 slag (AD-643(2))	Excess	0.26

* Mixture 3-B(2) included cube and bar specimens for strength and restrained expansion as well as vials for XRD examination; the other mixtures included only vial specimens for XRD examination.

Table 1B

Chemical and Physical Data for Blended

Type H Cement RC-BCHSR

Chemical Data	Type of Analysis, % Wet	Instrumental*
CaO, %	64.02	64.16 EDTA
SiO_2	22.48	22.34 Gravimetric
Al_2O_3	3.70**	3.73 AA
Fe_2O_3	3.82	3.78 AA
MgO	2.17	1.98 AA
SO_3	2.12	
Loss on Ignition, %	0.53	
Alkalies - Total as Na_2O, %		0.56
Na_2O, %		0.17 AA
K_2O , %		0.58 AA
Insoluble Residue, %	0.15	
TiO_2	0.21 AA	
P_2O_5, %	0.33 PE	
Mn_2O_3, %	0.04 AA	
BaO, %	0.09 PE	
SrO, %	0.15 PE	
Calculated Compounds	**Amount, %**	
C_3S, %	53	55
C_3A, %	3	3
C_2S, %	24	23
$C_3A + C_3S$, %	57	58
C_4AF, %	12	12
$C_4AF + 2\ C_3A$, %	18	18
Physical Data		
Surface Area, m^2/kg	210	
Air Content, %	9.9	
Comp. Strength, psi		
3 d	1520	
7 d	2090	
28 d	2500	
90 d	3600	
Autoclave Exp., %	0.00	
Initial Set, hr/min	4:05	
Final Set, hr/min	7:15	

* PE = Plasma Emission, AA = Atomic Absorption-flame.

** Referee Al_2O_3 = Corrected for TiO_2 and P_2O_5.

Table 1C

Chemical and Physical Data for Natural Pozzolan AD-518

Chemical Data, %				
SiO_2	67.98			
Al_2O_3	17.40			
Fe_2O_3	5.49			
MgO	0.80			
SO_3	0.88			
CaO	2.28			
Moisture Content	1.37			
LOI, % (750° C)	1.58			
LOI, % (1000° C)				
TiO_2				
P_2O_5				
Mn_2O_3				
Cr_2O_3				
Chloride				
Alkalies	Water Soluble	Available (C-618)*	Acid Soluble	Total Alkali
Na_2O	0.02	0.18	0.16	2.11
K_2O	0.00	0.26	0.19	1.59
Total as Na_2O	0.02	0.35	0.28	3.16

Physical Data

Specific Gravity: 2.39

Surface Area: cm^2/cc: 26.760

Porosity: e: 0.668

Tests with portland cement cured @ 73.4 ± 3° F

Portland Cement Co.:	United			Citadel		
Location:	Artesia, MS			Birmingham, AL		
Cement No. & Type:	RC-688, I, LA			RC-705, II, LA, HH		
Autoclave Expansion, 20% Replacement, %	0.03			0.06		
Cement replacement by volume, %	0	30	60	0	30	60
Heat of Hydration, cal/gm						
7 days	84.8	75	59	67.7	60	46
28 days	96.5	86	68	78.8	72	61

(Continued)

Table 1C (Concluded)

Compressive Strength, psi						
3 days	2880	2710	1120	1700	1710	920
7 days	4080	3920	1880	2510	2480	1480
28 days	5320	6050	4010	4040	4930	3640
90 days	5860	6780	6350	5760	5540	4860
180 days	6050	7330	7240	5990	5620	5380
1 year		7690	7250		5880	5460
Water - Cement Ratio, by mass	0.485	0.485	0.532	0.485	0.485	0.532
Flow, %	111	51	50	122	62	62

* Pozzolanic Activity Index, ASTM C 618; With Lime @ 7 days, psi: 1960; With Portland Cement (RC-688) at 28 days, percent of Control: 98.

Table 1D

Chemical and Physical Properties of Slag AD-643(2)

Chemical Data, %		Test Method
SiO_2	34.62	ASTM C-114-80 Referee (NH_4Cl)
Al_2O_3	9.32	R_2O_3 - (Fe_2O_3 + P_2O_5 + TiO_2) Referee
Fe_2O_3	0.88	ASTM C-114 Referee Titration
CaO	41.70	ASTM C-114 Optional A Gravimetric
MgO	10.42	ASTM C-114, Referee Gravimetric
SO_3	0.45	ASTM C-114 Referee
Loss on Ignition	0.83	ASTM C-114-80: 43-44
Insoluble Residue	0.28	ASTM C-114
Alkalies:		
Total as Na_2O	0.45	ASTM C-114 A-A
Na_2O	0.21	
K_2O	0.36	
Available as Na_2O	0.25	ASTM C-311 A-A
Na_2O	0.12	
K_2O	0.19	
Water Soluble as Na_2O	0.05	ASTM C-114 A-A
Na_2O	0.02	
K_2O	0.04	
TiO_2	0.46	ASTM C-114 Rapid A-A
P_2O_5	0.01	ASTM C-114 Referee
Mn_2O_3	0.34	ASTM C-114 Referee A-A
Sulfide Sulfur	1.07	ASTM C-114 Referee
Fe	0	Magnetic Separation

Physical Data		Test Method and Remarks
Fineness		
No. 325 sieve, % retained:	2	ASTM C-430
A.P. Surface Area, m^2/kg:	558	ASTM C-204, e = 0.530
Density, mg/m^3:	2.94	ASTM C-188
Compressive Strength, psi		Cured as ASTM C-109 cubes
1 day	1,280	N.C. paste cubes
3 days	4,720	Slag + 2% KOH sol'n
7 days	6,490	
Compressive Strength at 28 days		Cured as ASTM C-311 sec. 30. N.C. paste, cubes.
RC-688(3) without slag, psi:	16,100	Slag, 35% by vol of cement
RC-688(3) with slag, psi:	15,260	Compressive strength of cubes in excess of 13,000 psi determined on Baldwin 450,000-lb capacity universal
RC-853(2) without slag, psi:	15,090	testing machine using the 100,000-lb range.
RC-853(2) with slag, psi:	14,200	
Normal Consistency, %		ASTM C-187
RC-688(3)	20.7	
RC-688(3) with slag	25.1	
RC-853(2)	20.9	
RC-853(2) with slag	24.9	
Slag + 2% KOH sol'n:	29.7	
Time of Set, Gillmore:	hr:min	ASTM C-266
Slag + 2% KOH sol'n, Initial:	3:30	
Final:	7:45	

Table 1E

Chemical and Physical Properties of

Silica Fume AD-536(3)*

Chemical Data, %	
$SiO_2 + Al_2O_3 + Fe_2O_3$	97.7
MgO	0.2
SO_3	0.3
Available Alkalies	0.5
Moisture Content	0.2
Loss on Ignition	0.7
Physical Data	
AP Fineness, cm^2/cm^3 at porosity e = 0.714	42,550
Amount Retained on 45-μm (No. 325) Sieve, %	0.4
Combined with Portland Cement RC-688**	
Water Requirement, % of Control: 98	
Pozzolanic Activity, % of Control: 140	
Control W/C: 0.484, Flow 114%	
Test Mix W/C: 0.528, Flow 64%	
Autoclave Expansion, %: -0.06	
Pozzolanic Activity With Lime, psi: 2050 (2-in. cubes)	

* Done in general accordance with ASTM C 311.

** P. C. alone at 28 days at 100° F, psi 5190.

Table 2A

Stability and Persistence of Ettringite and Gypsum in Cement, Plaster, and Water Mixture* by X-Ray Diffraction**

Age		Temperature, °C 23	50	75
24 hr	– Ettringite	4	--	--
	Gypsum	3	--	--
48 hr	– Ettringite	7	9	11
	Gypsum	6	5	12
7 day	– Ettringite	10	11	15
	Gypsum	4	12	7
14 day	– Ettringite	10	14	10
	Gypsum	8	7	10
21 day	– Ettringite	11	17	14
	Gypsum	8	10	12
28 day	– Ettringite	15	17	13
	Gypsum	11	5	11
56 day	– Ettringite	16	20	14
	Gypsum	11	14	13
90 day	– Ettringite	19	20	17
	Gypsum	9	14	20
180 day	– Ettringite	17	22	16
	Gypsum	12	13	15
270 day	– Ettringite	17	21	14
	Gypsum	7	7	8
365 day	– Ettringite	17	25	16
	Gypsum	16	11	9

* Cast 2 made 3 June 1982; water to cementitious solids ratio (w/s) was 0.32.

** Peak intensity values are in net chart units; 9.7- and 7.6-A peaks.

Table 3A

Stability and Persistene of Ettringite and Gypsum in Cement, Plaster, Slag, and Water Mixture* by X-Ray Diffraction**

Age		Temperature, °C 23	50	75
24 hr	- Ettringite	6	--	--
	Gypsum	6	--	--
48 hr	- Ettringite	8	10	9
	Gypsum	7	7	9
7 day	- Ettringite	10	16	18
	Gypsum	6	9	8
14 day	- Ettringite	14	20	17
	Gypsum	10	7	5
21 day	- Ettringite	20	23	24
	Gypsum	13	5	5
28 day	- Ettringite	18	24	23
	Gypsum	9	7	6
56 day	- Ettringite	22	25	24
	Gypsum	8	8	4
90 day	- Ettringite	21	22	24
	Gypsum	10	7	5
180 day	- Ettringite	26	25	24
	Gypsum	7	4	4
270 day	- Ettringite	20	24	28
	Gypsum	8	5	4
365 day	- Ettringite	23	27	28
	Gypsum	8	5	3

* Cast 3 made 7 June 1982; w/s was 0.28.

** Peak intensity values are in net chart units; 9.7- and 7.6-A peaks.

Table 4A

Stability and Persistence of Ettringite and Gypsum in Cement, Plaster, Fly Ash AD-629, and Water Mixture* by X-Ray Diffraction**

Age		Temperature, °C 23	50	75
24 hr	- Ettringite	6	--	--
	Gypsum	7	--	--
48 hr	- Ettringite	10	14	24
	Gypsum	6	9	14
7 day	- Ettringite	16	25	33
	Gypsum	20	8	10
14 day	- Ettringite	20	30	33
	Gypsum	8	7	4
21 day	- Ettringite	18	37	35
	Gypsum	11	7	6
28 day	- Ettringite	21	37	41
	Gypsum	12	7	7
56 day	- Ettringite	23	34	33
	Gypsum	12	9	2
90 day	- Ettringite	13	26	29
	Gypsum	6	4	nd†
180 day	- Ettringite	32	40	††
	Gypsum	11	10	††
270 day	- Ettringite	31	41	37
	Gypsum	11	8	nd
365 day	- Ettringite	33	44	37
	Gypsum	10	7	nd

* Cast 4 made 9 June 1982; w/s was 0.38.

** Peak intensity values are in net chart units; 9.7- and 7.6-A peaks.

† Not detected.

†† Ettringite present, gypsum gone; not run on linear scale.

Table 5A

Stability and Persistence of Ettringite and Gypsum in Cement, Plaster, Fly Ash AD-513, and Water Mixture* by X-Ray Diffraction**

Age		Temperature, °C 23	50	75
24 hr	- Ettringite	7	--	--
	Gypsum	24	--	--
48 hr	- Ettringite	11	17	27
	Gypsum	13	14	12
7 day	- Ettringite	15	17	39
	Gypsum	11	4	9
14 day	- Ettringite	18	39	52
	Gypsum	14	7	11
21 day	- Ettringite	23	49	57
	Gypsum	15	6	10
28 day	- Ettringite	26	56	60
	Gypsum	22	15	17
59 day	- Ettringite	35	62	61
	Gypsum	16	9	8
90 day	- Ettringite	37	55	51
	Gypsum	21	8	10
180 day	- Ettringite	44	63	53
	Gypsum	11	9	3
270 day	- Ettringite	48	65	54
	Gypsum	9	14	8
365 day	- Ettringite	53	62	63
	Gypsum	10	8	12

* Cast 5 made 11 June 1982; w/s was 0.48.

** Peak intensity values are in net chart units; 9.7- and 7.6-A peaks.

Table 6A

Stability and Persistence of Ettringite and Gypsum in Cement, Plaster, Fly Ash AD-513, and Water Mixture* by X-Ray Diffraction**

Age		Temperature, °C 23	50	75
24 hr	- Ettringite	8	--	--
	Gypsum	10	--	--
48 hr	- Ettringite	11	20	28
	Gypsum	9	18	14
7 day	- Ettringite	18	33	32
	Gypsum	11	11	12
14 day	- Ettringite	24	37	39
	Gypsum	13	13	16
20 day	- Ettringite	21	42	45
	Gypsum	16	14	10
28 day	- Ettringite	28	46	46
	Gypsum	14	17	11
56 day	- Ettringite	33	47	46
	Gypsum	21	10	11
90 day	- Ettringite	24	44	48
	Gypsum	12	11	11
180 day	- Ettringite	35	46	48
	Gypsum	16	8	nd†
270 day	- Ettringite	35	47	44
	Gypsum	15	7	3
365 day	- Ettringite	37	49	50
	Gypsum	12	8	Trace

* Cast 6 made 15 June 1982; w/s was 0.35.

** Peak intensity values are in net chart units; 9.7- and 7.6-A peaks.

† Not detected.

Table 7A

Stability and Persistence of Ettringite and Gypsum in Cement, Plaster, Slag, and Water Mixture* by X-Ray Diffraction**

Age		Temperature, °C 23	50	75
24 hr	- Ettringite	6	--	--
	Gypsum	2	--	--
48 hr	- Ettringite	10	11	12
	Gypsum	6	5	4
7 day	- Ettringite	14	19	21
	Gypsum	5	7	7
14 day	- Ettringite	17	25	25
	Gypsum	10	9	7
21 day	- Ettringite	19	26	26
	Gypsum	7	6	8
28 day	- Ettringite	18	24	26
	Gypsum	13	8	8
56 day	- Ettringite	22	26	29
	Gypsum	7	3	5
90 day	- Ettringite	19	26	26
	Gypsum	8	5	4
180 day	- Ettringite	25	30	32
	Gypsum	13	6	4
270 day	- Ettringite	25	28	31
	Gypsum	7	4	3
365 day	- Ettringite	26	32	31
	Gypsum	5	3	3

* Cast 7 made 17 June 1982; w/s was 0.36.

** Peak intensity values are in net chart units; 9.7- and 7.6-A peaks.

Table 8A

Stability and Persistence of Ettringite and Gypsum in Cement, Plaster, Natural Pozzolan, and Water Mixture* by X-Ray Diffraction**

Age		Temperature, °C	
	23	50	75
24 hr - Ettringite	7	--	--
Gypsum	5	--	--
48 hr - Ettringite	9	12	11
Gypsum	12	11	18
7 day - Ettringite	16	19	15
Gypsum	12	15	12
14 day - Ettringite	19	23	20
Gypsum	11	16	11
21 day - Ettringite	21	27	25
Gypsum	15	20	19
28 day - Ettringite	21	22	24
Gypsum	9	13	8
56 day - Ettringite	21	14	15
Gypsum	24	7	12
90 day - Ettringite	21	24	21
Gypsum	17	10	18
180 day - Ettringite	20	28	18
Gypsum	13	14	12
270 day - Ettringite	24	29	20
Gypsum	18	17	14
365 day - Ettringite	24	27	20
Gypsum	14	13	11

* Cast 8 made 21 June 1982; w/s was 0.43.

** Peak intensity values are in net chart units, 9.7- and 7.6-A peaks.

Table 9A

Stability and Persistence of Ettringite and Gypsum in Cement, Plaster, Fly Ash AD-628, and Water Mixture* by X-Ray Diffraction**

Age		Temperature, °C 23	50	75
24 hr	- Ettringite	5	--	--
	Gypsum	14	--	--
48 hr	- Ettringite	5	9	8
	Gypsum	15	9	23
7 day	- Ettringite	10	22	25
	Gypsum	13	13	21
14 day	- Ettringite	14	30	28
	Gypsum	26	25	19
21 day	- Ettringite	15	30	26
	Gypsum	25	11	19
28 day	- Ettringite	17	31	28
	Gypsum	16	22	17
56 day	- Ettringite	8	29	24
	Gypsum	6	13	10
90 day	- Ettringite	15	21	16
	Gypsum	13	11	7
180 day	- Ettringite	26	36	30
	Gypsum	22	19	Trace
270 day	- Ettringite	26	33	33
	Gypsum	21	14	Trace
365 day	- Ettringite	24	33	29
	Gypsum	18	9	3

* Cast 9 made 23 June 1982; w/s = 0.34.

** Peak intensity values are in net chart units; 9.7- and 7.6-A peaks.

Table 10A

Stability and Persistence of Ettringite and Gypsum in Cement, Plaster, Silica Fume, and Water Mixture* by X-Ray Diffraction**

Age		Temperature, °C 23	50	75
24 hr	- Ettringite	10	--	--
	Gypsum	2	--	--
48 hr	- Ettringite	12	10	nd†
	Gypsum	3	8	7
7 day	- Ettringite	10	9	nd
	Gypsum	3	6	8
14 day	- Ettringite	13	9	4
	Gypsum	5	9	9
21 day	- Ettringite	12	8	Trace
	Gypsum	9	9	6
28 day	- Ettringite	12	8	Trace
	Gypsum	10	10	7
56 day	- Ettringite	11	7	nd
	Gypsum	8	7	7
90 day	- Ettringite	8	7	nd
	Gypsum	4	9	8
180 day	- Ettringite	13	7	nd
	Gypsum	8	8	7
270 day	- Ettringite	10	7	nd
	Gypsum	5	8	11
365 day	- Ettringite	13	7	nd
	Gypsum	10	7	10

* Cast 10 made 25 June 1982; w/s was 0.32.

** Peak intensity values are in net chart units; 9.7- and 7.6-A peaks.

† Not detected; there was sometimes a trace of ettringite in the slow pattern.

Table 11A

Stability and Persistence of Ettringite and Gypsum in Cement, Plaster, Natural Pozzolan, and Water Mixture* by X-Ray Diffraction**

Age		Temperature, °C 23	50	75
24 hr	- Ettringite	8	--	--
	Gypsum	5	--	--
48 hr	- Ettringite	10	13	14
	Gypsum	7	12	14
7 day	- Ettringite	16	19	19
	Gypsum	13	24	17
14 day	- Ettringite	19	21	20
	Gypsum	15	20	20
21 day	- Ettringite	20	22	20
	Gypsum	17	14	16
28 day	- Ettringite	20	19	19
	Gypsum	12	9	16
56 day	- Ettringite	22	24	19
	Gypsum	13	15	17
90 day	- Ettringite	15	22	19
	Gypsum	19	10	14
180 day	- Ettringite	21	23	21
	Gypsum	14	10	19
270 day	- Ettringite	20	25	22
	Gypsum	17	14	15
365 day	- Ettringite	24	30	23
	Gypsum	15	13	11

* Cast 11 made 29 June 1982; w/s was 0.36.

** Peak intensity values are in net chart units; 9.7- and 7.6-A peaks.

Table 12A

Stability and Persistence of Ettringite and Gypsum in Cement, Plaster, and Water Mixture* by X-Ray Diffraction**

Age		Temperature, °C 23	75	100
24 hr	- Ettringite	5	--	--
	Gypsum	3	--	--
48 hr	- Ettringite	8	10	Trace
	Gypsum	3	3	nd†
7 day	- Ettringite	10	12	Trace
	Gypsum	4	4	nd
14 day	- Ettringite	10	14	nd
	Gypsum	4	4	nd
21 day	- Ettringite	13	13	nd
	Gypsum	3	4	nd
28 day	- Ettringite	12	13	nd
	Gypsum	4	2	nd
56 day	- Ettringite	11	13	nd
	Gypsum	Trace	Trace	nd
90 day	- Ettringite	12	12	nd
	Gypsum	4	nd	nd
180 day	- Ettringite	14	14	nd
	Gypsum	2	nd	nd
270 day	- Ettringite	13	14	4
	Gypsum	3	nd	nd
365 day	- Ettringite	16	14	nd
	Gypsum	3	Trace	nd

* Cast 12 made 1 July 1982; w/s was 0.22.

** Peak intensity values are in net chart units; 9.7- and 7.6-A peaks.

† Not detected.

Table 13A

Stability and Persistence of Ettringite and Gypsum in Cement, Plaster, Fly Ash AD-629, and Water Mixture* by X-Ray Diffraction**

Age		Temperature, °C		
		23	75	100
24 hr	- Ettringite	11	--	--
	Gypsum	16	--	--
48 hr	- Ettringite	14	29	18
	Gypsum	11	11	3
7 day	- Ettringite	15	42	Trace
	Gypsum	11	9	nd†
14 day	- Ettringite	17	48	3
	Gypsum	10	9	nd
21 day	- Ettringite	22	48	nd
	Gypsum	7	4	nd
28 day	- Ettringite	23	44	nd
	Gypsum	10	4	nd
56 day	- Ettringite	33	43	nd
	Gypsum	6	4	nd
90 day	- Ettringite	33	43	nd
	Gypsum	7	3	nd
180 day	- Ettringite	37	45	nd
	Gypsum	13	nd	nd
270 day	- Ettringite	45	52	nd
	Gypsum	9	nd	nd
365 day	- Ettringite	48	53	Trace
	Gypsum	7	2	nd

* Cast 13 made 5 July 1982; w/s was 0.43.

** Peak intensity values are in net chart units; 9.7- and 7.6-A peaks.

† Not detected.

Table 14A

Stability and Persistence of Ettringite and Gypsum in Cement, Plaster, Silica Fume, and Water Mixture* by X-Ray Diffraction**

Age		Temperature, °C 23	75	100
24 hr	- Ettringite	10	--	--
	Gypsum	3	--	--
48 hr	- Ettringite	13	3	nd†
	Gypsum	3	11	8
6 day	- Ettringite	10	nd	nd
	Gypsum	6	8	11
13 day	- Ettringite	13	nd	nd
	Gypsum	6	6	9
20 day	- Ettringite	12	Trace	nd
	Gypsum	7	10	6
28 day	- Ettringite	10	nd	nd
	Gypsum	5	10	6
56 day	- Ettringite	11	2	nd
	Gypsum	9	9	5
90 day	- Ettringite	12	nd	nd
	Gypsum	10	7	10
180 day	- Ettringite	11	nd	nd
	Gypsum	8	9	2
270 day	- Ettringite	10	nd	nd
	Gypsum	8	10	nd
365 day	- Ettringite	††	nd	nd
	Gypsum	††	9	nd

* Cast 14 made 8 July 1982; w/s was 0.30.

** Peak intensity values are in net chart units; 9.7- and 7.6-A peaks.

† Not detected.

†† Present but not quantified on linear scale.

Table 16A

Stability and Persistence of Ettringite and Gypsum in Cement, Plaster, Fly Ash AD-628, and Water Mixture* by X-Ray Diffraction**

Age		Temperature, °C		
		23	75	100
24 hr	Ettringite	6	--	--
	Gypsum	31	--	--
48 hr	Ettringite	7	12	9
	Gypsum	18	17	18
7 day	Ettringite	8	25	nd†
	Gypsum	22	16	nd
14 day	Ettringite	11	32	nd
	Gypsum	26	15	Trace
21 day	Ettringite	14	30	nd
	Gypsum	20	14	nd
28 day	Ettringite	15	44	nd
	Gypsum	13	23	2
57 day	Ettringite	11	29	nd
	Gypsum	14	10	nd
90 day	Ettringite	21	42	nd
	Gypsum	30	19	nd
180 day	Ettringite	28	40	nd
	Gypsum	24	13	nd
270 day	Ettringite	29	44	nd
	Gypsum	19	17	nd
365 day	Ettringite	32	††	nd
	Gypsum	30	††	nd

* Cast 16 made 13 July 1982; w/s was 0.46.

** Peak intensity values are in net chart units; 9.7- and 7.6-A peaks.

† Not detected.

†† Not examined by error.

Table 17A

Stability and Persistence of Ettringite and Gypsum in Cement, Plaster, and Water Mixture* by X-Ray Diffraction**

Age		Temperature, °C 23	50	75	100
24 hr	- Ettringite	5	--	--	--
	Gypsum	2	--	--	--
48 hr	- Ettringite	7	10	10	9
	Gypsum	5	6	4	7
7 day	- Ettringite	9	13	13	6
	Gypsum	4	4	4	nd†
14 day	- Ettringite	13	17	16	Trace
	Gypsum	3	3	4	nd
21 day	- Ettringite	13	15	14	nd
	Gypsum	2	2	3	nd
28 day	- Ettringite	13	18	18	nd
	Gypsum	1	1	2	nd
56 day	- Ettringite	13	16	14	nd
	Gypsum	3	2	2	nd
90 day	- Ettringite	16	20	17	nd
	Gypsum	3	3	nd	nd
180 day	- Ettringite	13	21	15	?
	Gypsum	2	nd	nd	nd
270 day	- Ettringite	16	19	15	nd
	Gypsum	Trace	nd	nd	nd
365 day	- Ettringite	20	25	20	nd
	Gypsum	Trace	nd	nd	nd

* Cast 17 made 15 July 1982; w/s was 0.34.

** Peak intensity values are in net chart units; 9.7- and 7.6-A peaks.

† Not detected.

Table 2B

Compressive Strengths of Paste Cubes at Different Temperatures and Ages

Temperature °C	Control Mixture *	Compressive Strengths (psi) at Ages Shown Below, days										
		1	2	7	14	21	28	56	90	180	270	365
23	Avg**	4180	5010	6690	7890	8460	***	--	--	--	--	--
	Test Mixture †											
	Avg**	2240	2730	3950	4390	5350	6040	6930	7,570	5,510	9,230	12,340
50	Avg**	nd††	3220	5650	6450	7560	8000	9220	10,140	10,890	10,250	8,770
75	Avg**	nd	3720	6220	6780	7320	6760	8760	6,720	7,630	8,460	7,810

* Normal consistency paste of cement and plaster.

** Each value for the control mixture and the 1- and 2-day ages for the test mixture is the average of three 2- by 2- by 2-in. cubes; values for later ages of the test mixture are the averages for two cubes and single cubes beginning at 56 days through 180 days; the 270- and 365-day values are averages for three cubes.

*** All cubes used at 21 days.

† Cast 2 made 3 June 1982; cement, plaster, and high water content.

†† Not determined.

Table 3B

Compressive Strengths of Paste Cubes at Different Temperatures and Ages

Temperature °C	Control Mixture *	Compressive Strengths (psi) at Ages Shown Below, days										
		1	2	7	14	21	28	56	90	135	180	365
23	Avg**	5670	8590	8370	9,600	11,940	12,120	***	--	--	--	--
	Test Mixture †											
	Avg**	2400	3320	5190	6,350	7,930	8,180	10,020	8,240	12,260	11,850	13,400
50	Avg**	nd††	5220	8140	9,620	8,780	10,380	11,640	12,450	12,780	11,810	13,340
75	Avg**	nd	6180	9860	11,270	12,940	12,920	14,830	12,260	15,560	15,260	14,950

* Normal consistency paste of cement and plaster; same as Cast 12.

** Each value for the control mixture through 21 days and the 1- and 2-day ages for the test mixture is the average of three 2- by 2- by 2-in. cubes; values for later ages for the test mixture are averages of two cubes through 28 days and single cubes through 180 days; the final values are averages for three cubes.

*** All cubes used at 28 days.

† Cast 3 made 7 June 1982; normal consistency mixture of cement, plaster, and slag.

†† Not determined.

Table 4B

Compressive Strengths of Paste Cubes at Different Temperatures and Ages

Temperature °C	Control Mixture *	Compressive Strengths at Ages Shown Below, days											
		1	2	7	14	21	28	56	90	135	180	270	365
23	Avg**	6270	6850	8,670	9,180	10,060	10,400	***	--	--	--	--	--
	Test Mixture †												
	Avg**	2840	3430	5,070	5,940	6,390	7,360	7,720	8,000	9,800	10,970	10,990	10,850
50	Avg**	n.d.††	4610	8,310	9,600	11,190	10,970	12,280	10,020	13,230	12,120	12,590	13,670
75	Avg**	n.d.	6640	10,750	12,110	13,950	13,970	14,260	14,570	12,280	12,210	13,920	15,230

* Normal consistency paste of cement and plaster; same as cast 12.

** Each value for the control mixture is the average for three cubes. Values for the test mixture are averages for two 2- by 2- by 2-in. cubes through 28 days, single cubes through 180 days, and averages for three cubes at 270 and 365 days.

*** All cubes used at 28 days.

† Cast 4 made 9 June 1982; normal consistency mixture of cement, plaster, and fly ash AD-629.

†† Not determined.

Table 5B

Compressive Strengths of Paste Cubes at Different Temperatures and Ages

Temperature °C	Control Mixture *	Compressive Strengths (psi) at Ages Shown Below, days										
		1	2	7	14	21	28	56	90	180	270	365
23	Avg**	6170	7470	9200	10,080	10,670	11,130	***	--	--	--	--
	Test Mixture †											
	Avg**	540	790	1240	1,560	2,020	2,140	3190	3420	4140	4640	†††
50	Avg**	nd††	1170	3080	3,900	5,010	5,510	6410	5740	6450	6490	7100
75	Avg**	nd	2350	5150	5,270	5,620	6,320	6700	7680	6640	7570	6980

* Normal consistency paste of cement and plaster; same as Cast 12.

** Each value for the control mixture is the average of three 2- by 2- by 2-in. cubes; each value for the test mixture is the average of two cubes through 21 days; single cubes through 180 days; three cubes at 270 days; two cubes at 365 days.

*** All cubes used at 28 days.

† Cast 5 made 11 June 1982; cement, plaster, fly ash AD-513, and high water.

†† Not determined.

††† All remaining cubes tested at 270 days.

Table 6B

Compressive Strengths of Paste Cubes at Different Temperatures and Ages

Temperature ° C	Control Mixture *	Compressive Strengths at Ages Shown Below, days											
		1	2	7	14	21	28	56	90	135	180	270	365
23	Avg**	6240	7650	9020	10,700	10,980	11,150	***	--	--	--	--	--
	Test Mixture †												
	Avg**	1370	2090	2970	3,930	4,510	5,150	6,440	6,000	7,400	9,210	9,080	7,900
50	Avg**	n.d.††	3040	7130	8,130	9,230	9,290	10,350	9,040	9,400	9,450	11,610	11,240
75	Avg**	n.d.	4950	9150	8,950	10,620	10,760	12,500	8,030	9,880	10,000	10,400	9,830

* Normal consistency paste of cement and plaster; same as cast 12.

** Each value for the control mixture is the average of three 2- by 2- by 2-in. cubes; values for the test mixture are the averages for two cubes through 21 days; single cubes through 180 days; two cubes at 270 days; and three cubes at 365 days.

*** All cubes used at 28 days.

† Cast 6 made 15 June 1982; normal consistency mixture of cement, plaster, and fly ash AD-513.

†† Not determined.

Table 7B

Compressive Strengths of Paste Cubes at Different Temperatures and Ages

Temperature °C	Control Mixture *	Compressive Strengths at Ages Shown Below, days											
		1	2	7	14	21	28	56	90	135	180	270	365
23	Avg**	6720	7990	9710	10,920	11,080	11,670	***	--	--	--	--	--
	Test Mixture †												
	Avg**	1500	2200	3790	4,780	5,950	6,540	7,160	8,520	7,990	9,260	7,980	5,360
50	Avg**	n.d.††	3370	6280	8,050	8,720	8,870	9,680	10,970	10,210	12,540	12,500	12,390
75	Avg**	n.d.	2950	8290	9,650	9,200	7,570	9,940	10,880	7,440	8,060	10,260	11,040

* Normal consistency paste of cement and plaster; same as cast 12.

** Each value for the control mixture is the average of three 2- by 2- by 2-in. cubes; values for the test mixture are the averages for two cubes through 21 days; single cubes through 180 days; two cubes at 270 days; and three cubes at 365 days.

*** All cubes used at 28 days.

† Cast 7 made 17 June 1982; cement, plaster, slag, and high water.

†† Not determined.

Table 8B

Compressive Strengths of Paste Cubes at Different Temperatures and Ages

Temperature °C	Control Mixture *	Compressive Strengths at Ages Shown Below, days											
		1	2	7	14	21	28	56	90	135	180	270	365
23	Avg**	6060	7690	9160	9860	11,170	11,270	***	--	--	--	--	--
	Test Mixture †												
	Avg**	830	1290	2710	3340	3,750	4,460	5030	4830	4770	4850	6300	6480
50	Avg**	n.d.††	2660	4280	4780	5,200	5,110	5690	5540	7310	6180	7340	7400
75	Avg**	n.d.	3020	4350	5080	5,800	5,760	6210	5660	5890	7410	8510	8640

* Normal consistency paste of cement and plaster; same as cast 12.

** Each value for the control mixture is the average of three 2- by 2- by 2-in. cubes; values for the test mixture are the averages for two cubes through 21 days; single cubes through 180 days; three cubes at 270 and 365 days.

*** All cubes used at 28 days.

† Cast 8 made 21 June 1982; cement, plaster, natural pozzolan, and high water.

†† Not determined.

Table 9B

Compressive Strengths of Paste Cubes at Different Temperatures and Ages

Temperature °C	Control Mixture *	Compressive Strengths at Ages Shown Below, days											
		1	2	7	14	21	28	56	90	135	180	270	365
23	Avg**	5920	7220	8840	9840	10,580	10,950	***	--	--	--	--	--
	Test Mixture †												
	Avg**	1530	2020	2980	3680	4,160	5,020	5,520	6700	7,480	7,410	9,150	8,000
50	Avg**	n.d.††	2450	5770	8090	8,740	10,170	9,740	9830	10,290	10,100	11,480	13,580
75	Avg**	n.d.	3900	8140	9670	10,560	10,380	11,050	6700	9,080	9,720	9,850	11,790

* Normal consistency paste of cement and plaster; same as cast 12.

** Each value for the control mixture is the average of three 2- by 2- by 2-in. cubes; values for the test mixture are the averages for two cubes through 14 days; single cubes through 180 days; three cubes at 270 days; two cubes at 365 days.

*** All cubes used at 28 days.

† Cast 9 made 23 June 1982; normal consistency mixture of cement, plaster, and fly ash AD-628.

†† Not determined.

Table 10B

Compressive Strengths of Paste Cubes at Different Temperatures and Ages

Temperature °C	Control Mixture *	Compressive Strengths at Ages Shown Below, days											
		1	2	7	14	21	28	56	90	135	180	270	365
23	Avg**	6600	7740	9110	10,660	10,960	10,650	***	--	--	--	--	--
	Test Mixture †												
	Avg**	1980	2680	6640	6,240	7,540	8,780	9,060	8440	10,250	9,050	8,310	†††
50	Avg**	n.d.††	7380	7180	9,180	10,810	10,650	9,850	9370	9,910	10,310	10,590	†††
75	Avg**	n.d.	8710	9800	12,110	12,120	10,180	10,610	7480	7,590	7,990	8,680	†††

* Normal consistency paste of cement and plaster; same as cast 12.

** Each value for the control mixture is the average for three 2- by 2- by 2-in. cubes through 21 days and then two cubes; values for the test mixture are the averages for two cubes through 14 days; single cubes at 21 days, two cubes at 28 days; single cubes through 180 days; three or four cubes at 270 days.

*** All cubes used at 28 days.

† Cast 10 cast 25 June 1982; cement, plaster, silica fume, and high water.

†† Not determined.

††† No cubes to test.

Table 11B

Compressive Strengths of Paste Cubes at Different Temperatures and Ages

Temperature °C	Control Mixture *	Compressive Strengths at Ages Shown Below, days											
		1	2	7	14	21	28	56	90	135	180	270	365
23	Avg**	6970	8380	10,100	11,040	12,080	12,460	***	--	--	--	--	--
	Test Mixture †												
	Avg**	1550	2290	4,000	5,100	5,880	6,170	7,150	5980	6,830	6,250	7,080	†††
50	Avg**	n.d.††	3710	5,410	5,680	6,310	6,540	7,850	7630	10,050	9,420	10,960	†††
75	Avg**	n.d.	4700	6,410	7,150	8,720	8,920	10,980	9840	10,570	11,380	11,190	†††

* Normal consistency paste of cement and plaster; same as cast 12.

** Each value for the control mixture is the average for three 2- by 2- by 2-in. cubes; values for the test mixture are the averages for two cubes through 21 days; single cubes through 135 days; two cubes at 180 days; three cubes at 270 days.

*** All cubes used at 28 days.

† Cast 29 June 1982; cement, plaster, natural pozzolan, and water for normal consistency.

†† Not determined.

††† No cubes remained.

Table 12B

Compressive Strengths of Paste Cubes at Different Temperatures and Ages

Temperature °C	Control Mixture *	Compressive Strengths at Ages Shown Below, days											
		1	2	7	14	21	28	56	90	135	180	270	365
23	Avg**	7260	8,240	9,100	10,910	11,390	11,220	***	--	--	--	--	--
	Test Mixture †												
	Avg**	6090	8,290	9,640	9,480	11,040	12,510	12,290	12,250	12,000	11,490	13,190	13,500
75	Avg**	n.d.††	11,030	12,660	12,220	12,490	14,280	12,570	14,920	14,510	14,190	15,400	14,650
100†††	Avg**	n.d.	10,620	10,950	13,320	14,960	11,750	15,500	11,400	11,790	12,230	11,910	8,400

* Normal consistency paste of cement and plaster; same as this test mixture.

** Each value for the control mixture is the average for three 2- by 2- by 2-in. cubes; values for the test mixtures are averages for two cubes through 7 days; single cubes at 14 days, then two cubes at 21 days; single cubes thereafter through 135 days, two cubes at 180 days; three cubes at 270 and 365 days.

*** All cubes used at 28 days.

† Cast 1 July 1982; cement, plaster, and water for normal consistency.

†† Not determined.

††† No heat for one week at about 135-days age and again for few days at about 270 days.

Table 13B

Compressive Strengths of Paste Cubes at Different Temperatures and Ages

Temperature °C	Control Mixture *	Compressive Strengths at Ages Shown Below, days											
		1	2	7	14	21	28	56	90	135	180	270	365
23	Avg**	7210	9140	11,600	11,470	12,180	12,190	***	--	--	--	--	--
	Test Mixture †												
	Avg**	780	1300	2,350	2,190	2,970	3,380	3960	3890	4480	4780	5150	6190
75	Avg**	n.d.††	3250	7,120	8,700	7,950	9,160	9290	7050	8460	8170	7750	7430
100	Avg**	n.d.	2590	2,760	2,860	2,890	8,070	2810	2330	2550	2340	3140	2100

* Normal consistency paste of cement and plaster; same as cast 12.

** Each value for the control mixture is the average for three 2- by 2- by 2-in. cubes; values for the test mixture are averages for two cubes with single cubes at 14 days and again after 28 days through 180 days; two to four cubes, usually three, at 270 and 365 days.

*** All cubes used at 28 days.

† Cast 5 July 1982; cement, plaster, fly ash AD-629, and excess water.

†† Not determined.

Table 14B

Compressive Strengths of Paste Cubes at Different Temperatures and Ages

Temperature °C	Control Mixture *	Compressive Strengths at Ages Shown Below, days											
		1	2	7	14	21	28	56	90	135	180	270	365
23	Avg**	7300	8,480	9,770	11,010	10,770	11,730	***	--	--	--	--	--
	Test Mixture †												
	Avg**	2870	3,790	5,270	7,070	6,840	10,810	8980	8,810	8380	10,740	8,820	6290
75	Avg**	n.d.††	6,150	7,970	10,250	7,910	10,380	8290	11,310	9480	6,380	12,800	9650
100	Avg**	n.d.	10,780	14,850	7,480	10,960	7,490	8050	11,590	2900	7,980	5,050	†††

* Normal consistency paste of cement and plaster; same as cast 12.

** Each value for the control mixture is the average for three 2- by 2- by 2-in. cubes; values for the test mixture are averages for two cubes through 180 days; usually three cubes at 270 and 365 days.

*** All cubes used at 28 days.

† Cast 8 July 1982; cement, plaster, silica fume, and water for normal consistency.

†† Not determined.

††† No cubes remained.

Table 16B

Compressive Strengths of Paste Cubes at Different Temperatures and Ages

Temperature ° C	Control Mixture *	Compressive Strengths at Ages Shown Below, days											
		1	2	7	14	21	28	56	90	135	180	270	365
23	Avg**	6360	7840	9980	10,300	11,120	10,820	***	--	--	--	--	--
	Test Mixture †												
	Avg**	560	840	1180	1,550	1,790	1,980	2580	2850	3900	3480	4460	5790
75	Avg**	n.d.††	1480	4960	5,490	6,160	6,210	7170	6860	7850	6880	7630	8010
100	Avg**	n.d.	2020	2780	3,100	3,130	3,360	3230	3610	3420	3130	3500	3270

* Normal consistency paste of cement and plaster; same as cast 12.

** Each value for the control mixture is the average for three 2- by 2- by 2-in. cubes; values for the test mixture are averages for two cubes through 180 days; three or four cubes at 270 and 365 days.

*** All cubes broken at 28 days.

† Cast 13 July 1982; cement, plaster, fly ash AD-628, and excess water.

†† Not determined.

Table 17B

Compressive Strengths of Paste Cubes at Different Temperatures and Ages

Temperature °C	Control Mixture *	Compressive Strengths at Ages Shown Below, days											
		1	2	7	14	21	28	56	90	135	180	270	365
23	Avg**	6480	8500	10,040	10,240	10,580	12,190	***	--	--	--	--	--
	Test Mixture †												
	Avg**	1710	2520	4,040	5,270	5,880	6,070	6970	7690	8950	8750	8370	†††
75	Avg**	n.d.††	4050	4,300	7,770	8,290	7,980	8360	7750	8820	7990	8600	†††
100	Avg**	n.d.	4660	4,660	6,090	5,440	5,870	4440	5020	4950	5060	5950	†††

* Normal consistency paste of cement and plaster; same as cast 12.

** Each value for the control mixture is the average for three 2- by 2- by 2-in. cubes; values for the test mixture are averages for two cubes through 180 days; three cubes at 270 days.

*** All cubes used at 28 days.

† Cast 15 July 1982; cement, plaster, and excess water.

†† Not determined.

††† No cubes remained.

Table 18B

Compressive Strengths of Paste Cubes at Different Temperatures and Ages

Temperature °C	Control Mixture *	Compressive Strengths at Ages Shown Below, days										
		1	2	7	14	21	28	56	135	180	270	365
23	Avg**	5860	7700	9840	10,970	10,760	11,840	***	--	--	--	--
	Test Mixture †											
	Avg**	700	960	1130	2,610	2,030	3,510	3980	4460	4210	4270	4040
75	Avg**	††	900	1690	2,110	2,020	2,100	2270	2210	2160	1870	2440
100	Avg**	††	1260	1510	1,780	1,480	1,820	1850	2120	2100	2360	2240

* Normal consistency paste of cement and plaster; same as Cast 12.

** Each value for the control mixture is the average of three 2- by 2- by 2-in. cubes; values for the test mixture are the averages for two cubes through 28 days; single cubes at 56 days; two to four, usually two cubes at 135 and 180 days; and three cubes at 270 and 365 days.

*** All cubes used at 28 days.

† Cast 18 made 10 August 1982; excess water mixture of cement, Na_2SO_4, and fly ash AD-628. Compare with Mixture 16.

†† Not determined.

Table 2C

Restrained Expansion of Bars Cured 24 hr at 23^{o} C and Then Stored at 23^{o} C and Other Temperatures

Test Mixture No. 2*	Restrained Expansion of Single Bars at Ages Shown Below, %											
	1 (23^{o} C)	Temperature ^{o}C	2	7	14	21	28	56	90	180	270	365
Bar 1	-0.009**	23		0.014	0.013	0.025	0.024	0.019	0.029	0.035	0.049	-0.003
Bar 2	0.007	50		0.067	0.071	0.064	0.064	0.074	0.096	0.122	0.127	0.099
Bar 3	0.000	75		0.067	0.117	0.020	0.011	-0.010	0.027	0.049	0.061	0.025

* Cement, plaster, and excess water.

** All values are positive unless preceded by a minus sign.

Table 3C

Restrained Expansion of Bars Cured 24 hr at 23° C and Then Stored at 23° C and Other Temperatures

Test Mixture No. 3*	Restrained Expansion of Single Bars at Ages Shown Below, %											
	1 (23° C)	Temperature °C	2	7	14	21	28	56	90	135	180	365
Bar 6	-0.004**	23	0.006	0.028	0.027	0.026	0.026	0.018	-0.001	0.057	0.063	0.044
Bar 9	0.003	50	0.016	0.056	0.046	0.050	0.058	0.039	0.068	0.128	0.148	0.142
Bar 11	-0.012	75	0.037	0.059	0.033	0.007	0.000	-0.021	0.077	0.072	0.103	0.133

* Cement, plaster, slag, and water for normal consistency.
** All values are positive unless preceded by a minus sign.

Table 4C

Restrained Expansion of Bars Cured 24 hr at 23° C and Then Stored at 23° C and Other Temperatures

Test Mixture No. 4 *	Restrained Expansion of Single Bars at Ages Shown Below, %													
	1 (23° C)	Temperature °C	2	7	14	21	28	56	90	135	180	270	365
Bar 4	0.009**	23	0.019	0.033	0.164	0.074	0.084	0.085	0.128	0.163	0.190	1.522	1.422
Bar 15	0.004	50	0.070	0.083	0.085	0.159	0.182	0.192	0.668	0.904	0.103	-0.098	-0.134
Bar 24	0.011	75	0.089	0.036	0.034	0.030	0.041	0.041	0.094	0.204	0.240	0.254	0.237

* Cement, plaster, fly ash AD-629, and water for normal consistency.

** All values are positive unless preceded by a minus sign.

Table 5C

Restrained Expansion of Bars Cured 24 hr at 23° C and Then Stored at 23° C and Other Temperatures

Test Mixture No. 5*	Restrained Expansion of Single Bars at Ages Shown Below, %											
	1 (23° C)	Temperature °C	2	7	14	21	28	56	90	180	270	365
Bar 13	0.003**	23	0.009	0.024	0.038	0.048	0.060	0.092	0.110	0.144	0.178	0.217
Bar 16	0.006	50	0.062	0.093	0.143	0.171	0.187	0.179	0.252	0.330	0.318	0.173
Bar 17	0.008	75	0.090	0.088	0.092	0.063	0.031	-0.003	0.035	0.040	0.124	†

* Cement, plaster, fly ash AD-513, and excess water.

** All values are positive unless preceded by a minus sign.

† Insert corroded; no reading made.

Table 6C

Restrained Expansion of Bars Cured 24 hr at 23° C and Then Stored at 23° C and Other Temperatures

Test Mixture No. 6 *	Restrained Expansion of Single Bars at Ages Shown Below, %												
	1 (23° C)	Temperature °C	2	7	14	21	28	56	90	135	180	270	365
Bar 19	n.d.**	23	0.010†	0.044	0.159	0.074	0.082	0.101	0.119	0.130	0.104	0.273	0.190
Bar 25	n.d.	50	0.023	0.067	0.173	0.231	0.258	0.318	0.474	0.562	0.626	0.681	0.710
Bar 28	n.d.	75	0.139	0.120	0.130	0.143	0.182	0.092	0.123	0.215	0.248	0.215	0.221

* Cement, plaster, fly ash AD-513, and water for normal consistency.

** Not determined.

† All values are positive unless preceded by a minus sign.

Table 7C

Restrained Expansion of Bars Cured 24 hr at 23° C and Then Stored at 23° C and Other Temperatures

Test Mixture No. 7 *	Restrained Expansion of Single Bars at Ages Shown Below, %												
	1 (23° C)	Temperature °C	2	7	14	21	28	56	90	135	180	270	365
Bar 14	0.002	23	0.011†	0.030	0.036	0.048	0.044	0.049	0.071	0.066	0.089	0.119	0.145
Bar 20	n.d.**	50	0.017	0.028	0.060	0.089	0.103	0.129	0.140	0.163	0.183	0.163	0.166
Bar 27	n.d.	75	0.038	0.064	0.072	0.070	0.032	0.038	0.105	0.122	0.136	0.009	0.134

* Cement, plaster, slag, and excess water.
** Not determined.
† All values are positive unless preceded by a minus sign.

Table 8C

Restrained Expansion of Bars Cured 24 hr at 23° C and Then Stored at 23° C and Other Temperatures

	Restrained Expansion of Single Bars at Ages Shown Below, %												
Test Mixture No. 8 *	1 (23° C)	Temperature °C	2	7	14	21	28	56	90	135	180	270	365
Bar 12	0.006**	23	0.009	0.009	0.014	0.012	0.009	0.010	0.010	0.009	0.030	0.055	0.031
Bar 18	0.011	50	0.017	0.020	0.050	0.048	0.056	0.092	0.113	0.132	0.141	0.155	0.126
Bar 22	0.005	75	0.013	0.052	0.064	0.039	0.012	-0.010	0.061	0.072	0.069	0.073	0.067

* Cement, plaster, natural pozzolan, and excess water.

** All values are positive unless preceded by a minus sign.

Table 9C

Restrained Expansion of Bars Cured 24 h at 23° C and Then Stored at 23° C and Other Temperatures

Test Mixture No. 9 *	Restrained Expansion of Single Bars at Ages Shown Below, %												
	1 (23° C)	Temperature °C	2	7	14	21	28	56	90	135	180	270	365
Bar 21	0.004	23	0.008	0.016	0.025	0.016	0.020	0.025	0.030	0.031	0.015	0.101	0.099
Bar 29	-0.005**	50	0.005	0.078	0.074	0.115	0.127	0.214	0.229	0.276	0.254	0.363	0.379
Bar 42	-0.005	75	0.016	0.057	0.073	0.041	0.032	0.107	0.278	0.190	0.142	0.199	0.193

* Cement, plaster, fly ash AD-628, and water for normal consistency.

** All values are positive unless preceded by a minus sign.

Table 10C

Restrained Expansion of Bars Cured 24 hr at 23° C and Then Stored at 23° C and Other Temperatures

	Restrained Expansion of Single Bars at Ages Shown Below, %												
Test Mixture No. 10*	1 (23° C)	Temperature °C	2	7	14	21	28	56	90	135	180	270	365
Bar 36	-0.010**	23	-0.012	0.022	-0.014	-0.051	-0.087	-0.004	-0.064	-0.016	-0.005	-0.005	-0.038
Bar 37	-0.010	50	-0.074	-0.013	-0.011	-0.010	-0.004	-0.006	-0.041	0.017	0.018	0.030	0.005
Bar 48	-0.004	75	-0.067	-0.023	0.002	0.001	0.018	0.002	0.023	0.078	0.075	0.062	0.016

* Cement, plaster, silica fume, and excess water.

** All values are positive unless preceded by a minus sign.

Table 11C

Restrained Expansion of Bars Cured 24 hr at 23° C and Then Stored at 23° C and Other Temperatures

Test Mixture No. 11*	Restrained Expansion of Single Bars at Ages Shown Below, %												
	1 (23° C)	Temperature $^{\circ}$C	2	7	14	21	28	56	90	135	180	270	365
Bar 29	-0.010**	23	-0.004	-0.007	-0.002	0.016	0.008	0.006	0.002	0.012	0.028	0.048	0.027
Bar 30	-0.007	50	0.011	0.021	0.026	0.022	0.048	0.127	0.115	0.144	0.142	0.223	0.139
Bar 32	-0.005	75	0.050	0.016	0.009	0.011	-0.001	0.083	0.158	0.053	0.064	0.055	0.032

* Cement, plaster, natural pozzolan, and water for normal consistency.
** All values are positive unless preceded by a minus sign.

Table 12C

Restrained Expansion of Bars Cured 24 hr at 23° C and Then Stored at 23° C and Other Temperatures

	Restrained Expansion of Single Bars at Ages Shown Below, %												
Test Mixture No. 12*	1 (23° C)	Temperature °C	2	7	14	21	28	56	90	135	180	270	365
Bar 69	-0.001**	23	-0.001	-0.002	-0.001	-0.003	-0.004	0.031	0.020	0.001	0.002	0.041	0.049
Bar 72	0.001	75	-0.009	0.002	-0.013	-0.019	-0.015	0.005	0.031	0.038	0.057	0.056	0.039
Bar 79	0.003	100	-0.001	0.008	0.006	0.005	0.014	0.016	0.007	0.016	0.027	0.008	0.016

* Cement, plaster, and water for normal consistency.

** All values are positive unless preceded by a minus sign.

Table 13C

Restrained Expansion of Bars Cured 24 hr at 23° C and Then Stored at 23° C and Other Temperatures

Test Mixture No. 13*	Restrained Expansion of Single Bars at Ages Shown Below, % 1 (23° C)	Temperature °C	2	7	14	21	28	56	90	135	180	270	365
Bar 31	0.002**	23	0.030	0.042	0.053	0.066	0.086	0.142	0.123	0.135	0.169	0.235	0.250
Bar 78	0.004	75	0.064	0.093	0.124	0.133	0.194	0.163	0.218	0.232	0.230	0.197	0.217
Bar 80	0.011	100	0.063	0.081	0.158	0.103	0.159	0.073	0.073	0.083	0.063	0.025	0.046

* Cement, plaster, fly ash AD-629, and excess water.

** All values are positive unless preceded by a minus sign.

Table 14C

Restrained Expansion of Bars Cured 24 hr at 23° C and Then Stored at 23° C and Other Temperatures

Test Mixture No. 14*	Restrained Expansion of Single Bars at Ages Shown Below, %												
	1 (23° C)	Temperature °C	2	7	14	21	28	56	90	135	180	270	365
Bar 40	0.002**	23	-0.004	Skipped	-0.055	-0.087	-0.105	-0.017	-0.008	-0.004	-0.010	-0.042	-0.001
Bar 57	-0.010	75	-0.055	-0.047	-0.044	-0.018	0.014	-0.031	-0.012	-0.002	-0.005	-0.012	-0.017
Bar 62	-0.002	100	-0.029	-0.016	-0.019	-0.009	-0.015	-0.009	0.003	0.063	0.053	0.000	0.051

* Cement, plaster, silica fume, and water for normal consistency.

** All values are positive unless preceded by a minus sign.

Table 16C

Restrained Expansion of Bars Cured 24 hr at 23° C and Then Stored at 23° C and Other Temperatures

Test Mixture No. 16*	Restrained Expansion of Single Bars at Ages Shown Below, %												
	1 (23° C)	Temperature °C	2	7	14	21	28	56	90	135	180	270	365
Bar 34	-0.012**	23	-0.004	-0.004	0.008	0.020	0.016	0.046	0.021	0.003	0.026	0.028	-0.049
Bar 52	0.090	75	0.019	0.046	0.093	0.112	0.137	0.136	0.137	0.147	0.156	0.131	0.128
Bar 63	-0.002	100	0.030	0.029	0.051	0.039	0.032	0.054	0.054	0.016	0.026	0.019	0.015

* Cement, plaster, fly ash AD-628, and excess water.

** All values are positive unless preceded by a minus sign.

Table 17C

Restrained Expansion of Bars Cured 24 hr at 23° C and Then Stored at 23° C and Other Temperatures

	Restrained Expansion of Single Bars at Ages Shown Below, %												
Test Mixture No. 17*	1 (23° C)	Temperature °C	2	7	14	21	28	56	90	135	180	270	365
Bar 58	0.004**	23	0.006	0.006	0.019	0.021	0.023	-0.084	-0.081	-0.100	-0.087	-0.052	-0.066
Bar 61	-0.001	75	0.011	-0.008	0.023	0.019	0.020	-0.031	0.031	0.041	0.041	0.042	0.017
Bar 71	0.001	100	0.005	0.133	0.017	0.027	0.014	0.032	0.232	0.119	0.199	0.280	0.020

* Cement, plaster, and water for normal consistency.

** All values are positive unless preceded by a minus sign.

Table 18C

Restrained Expansion of Bars Cured 24 hr at 23° C and Then Stored at 23° C and Other Temperatures

Test Mixture No. 18*	Restrained Expansion of Single Bars at Ages Shown Below, % 1 (23° C)	Temperature °C	2	7	14	21	28	56	135	180†	270	365
Bar 81	0.015**	23	0.030	0.046	0.041	0.042	0.033	0.022	0.030	-0.055	0.061	-0.014
Bar 82	0.004	75	0.018	0.044	0.037	0.052	0.041	0.062	0.087	-0.012	0.107	0.099
Bar 85	0.018	100	0.040	0.057	0.047	0.059	0.054	0.061	0.072	-0.030	0.085	0.048

* Cement, Na_2SO_4, fly ash AD-628, and excess water. Compare with Mixture 16 in Table 16C.

** All values are positive unless preceded by a minus sign.

† Bars 81 and 82 were falling apart; Bar 85 was dry.

Table 19

Stability and Persistence of Chloroaluminate and Ettringite in Cement, Salt, and Water Mixture* by X-Ray Diffraction**

Age		Temperature, °C	
	23°	130° (25 psi)††	170° (100 psi)***
24 hr - Ettringite	9	--	--
Chloroaluminate	nd†	--	--
48 hr - Ettringite	6	nd	nd
Chloroaluminate	nd	nd	nd
7 day - Ettringite	4	nd	nd
Chloroaluminate	nd	nd	nd
14 day - Ettringite	5	nd	nd
Chloroaluminate	Trace	nd	nd
28 day - Ettringite	9	nd	
Chloroaluminate	nd	nd	
56 day - Ettringite	9	nd	
Chloroaluminate	nd	nd	
90 day - Ettringite	6	nd	
Chloroaluminate	nd	nd	
150 day - Ettringite	6	nd	
Chloroaluminate	nd	nd	
1 year - Ettringite	6	nd	
Chloroaluminate	nd	nd	

* Cast 1-B made 31 August 1982, w/c ratio was 0.24.

** Peak intensity values are in net chart units; 9.7- and 7.8-A peaks.

*** Test had to be stopped after 14 days.

† Not detected.

†† Specimens kept at 23° C for 4 days between 14 and 28 days.

Table 20

Stability and Persistence of Chloroaluminate and Ettringite in Cement, Salt, Fly Ash AD-628, and Water Mixture* by X-Ray Diffraction**

Age	Temperature, °C		
	23°	130° (25 psi)††	170° (100 psi)***
24 hr - Ettringite	7	--	--
Chloroaluminate	nd†	--	--
48 hr - Ettringite	7	nd	nd
Chloroaluminate	2	Trace	nd
7 day - Ettringite	8	nd	nd
Chloroaluminate	5	nd	nd
14 day - Ettringite	7	nd	nd
Chloroaluminate	7	nd	nd
28 day - Ettringite	8	nd	
Chloroaluminate	8	nd	
56 day - Ettringite	9	nd	
Chloroaluminate	8	nd	
90 day - Ettringite	8	nd	
Chloroaluminate	10	Trace	
150 day - Ettringite	8	nd	
Chloroaluminate	7	nd	
1 year - Ettringite	7	nd	
Chloroaluminate	7	nd	

* Cast 2-B made 31 August 1982; w/c ratio was 0.29.
** Peak intensity values are in net chart units; 9.7- and 7.8-A peaks.
*** Test had to be stopped after 14 days.
† Not detected.
†† Specimens kept at 23° C for 4 days between 14 and 28 days.

Table 21

Stability and Persistence of Chloroaluminate and Ettringite in Cement, Salt, Fly Ash AD-513, and Water Mixture* by X-Ray Diffraction**

Age	Temperature, °C		
	23°	130° (25 psi)††	170° (100 psi)***
24 hr - Ettringite	nd†	--	--
Chloroaluminate	2	--	--
48 hr - Ettringite	nd	nd	nd
Chloroaluminate	5	9	nd
7 day - Ettringite	nd	nd	nd
Chloroaluminate	11	Trace	nd
14 day - Ettringite	nd	nd	nd
Chloroaluminate	20	Trace	nd
28 day - Ettringite	nd	nd	
Chloroaluminate	22	nd	
56 day - Ettringite	nd	nd	
Chloroaluminate	26	nd	
90 day - Ettringite	nd	nd	
Chloroaluminate	24	nd	
150 day - Ettringite	nd	nd	
Chloroaluminate	27	nd	
1 year - Ettringite	nd	nd	
Chloroaluminate	25	nd	

* Cast 3-B made 1 September 1982; w/c ratio was 0.30.
** Peak intensity values are in net chart units; 9.7- and 7.8-A peaks.
*** Test had to be stopped after 14 days.
† Not detected.
†† Specimens kept at 23° C for 4 days between 14 and 28 days.

Table 22

Stability and Persistence of Chloroaluminate and Ettringite in Cement, Salt, Fly Ash AD-513 and Water Mixture* by X-Ray Diffraction**

Age		Temperature, ^{o}C	
		23^{o}	170^{o} (100 psi)††
24 hr	Ettringite	nd†	--
	Chloroaluminate	3	--
48 hr	Ettringite	nd	nd
	Chloroaluminate	3	nd
7 day	Ettringite	nd	nd***
	Chloroaluminate	13	nd
14 day	Ettringite		
	Chloroaluminate		
21 day	Ettringite		
	Chloroaluminate		
28 day	Ettringite		
	Chloroaluminate		

* Cast 3-B(2) made 17 September 1982; this was a repeat; w/c ratio was 0.30.

** Peak intensity values are in net chart units; 9.7- and 7.8-A peaks.

*** Temperature had exceeded 200^{o} C for short time.

† Not detected.

†† Test had to be stopped after 48 hr; tests of low temperature specimens were also stopped.

Table 23

Stability and Persistence of Chloroaluminate and Ettringite in Cement, Salt, Slag, and Water Mixture* by X-Ray Diffraction**

Age	Temperature, °C		
	23°	130° (25 psi)††	170° (100 psi)***
24 hr - Ettringite	2	--	--
Chloroaluminate	Trace	--	--
48 hr - Ettringite	2	nd†	nd
Chloroaluminate	5	5	6
7 day - Ettringite	2	nd	nd
Chloroaluminate	9	5	nd
14 day - Ettringite	Trace	nd	nd
Chloroaluminate	13	6	Trace
28 day - Ettringite	3	nd	
Chloroaluminate	12	7	
56 day - Ettringite	Trace	nd	
Chloroaluminate	13	5	
90 day - Ettringite	4	nd	
Chloroaluminate	14	7	
150 day - Ettringite	nd	nd	
Chloroaluminate	12	Trace	
1 year - Ettringite	nd	nd	
Chloroaluminate	15	Trace	

* Cast 4-B made 1 September 1982; w/c ratio was 0.26.
** Peak intensity values are in net chart units; 9.7- and 7.8-A peaks.
*** Test had to be stopped after 14 days.
† Not detected.
†† Specimens kept at 23° C for 4 days between 14 and 28 days.

Table 24

Compressive Strengths of Paste Cubes at Different Temperatures and Ages*

Temperature, °C	Compressive Strength at Ages Shown Below, days**	
	4	7†
23	2720	3490
170	3080	2590††

* Mixture 3B(2) made of cement, fly ash AD-513, salt, and water (w/s, 0.30).

** Single cubes at 23° C; average of two cubes at 170° C.

† Test had to be stopped after 7 days.

†† Temperature had risen to about 200° C and pressure had dropped to about 20 psi before cubes were broken.

Table 25

Restrained Expansion of Bars Cured 24 hr at 23° C and Then Stored at 23° C and 170° C

Mixture 3B(2)*	Temperature, °C	Restrained Expansion (ΔL) of Bars at Ages Shown, %**	
		2	7†
Bar 1	23	0.102	0.020
Bar 2		0.104	0.011
Average		0.103	0.016
Bar 3	170	0.124	-0.026††

* Mixture 3B(2) made of cement, fly ash AD-513, salt, and water (w/s, 0.30).

** % ΔL = (test length - initial length) x (100/10).

† Test had to be stopped after 7 days.

†† Values are positive unless preceded by a minus sign.

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